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Effects of Microstructure and Adhesion on Performance of Sputter-Deposited MoS_2 Solid Lubricant Coatings

Prepared by

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Selection of a lubricant for a given application (use) is determined by a number of factors. Analogous to fluid lubricants, where proper base stock, additive package, and formulation are selected for the application, choice of dry lubricants requires consideration of the appropriate deposition procedure, film composition, film structure, and substrate surface preparation procedure. The relationship between film physical and mechanical properties of MoS_2 produced by rf, dc, and rf magnetron sputtering is reviewed. We propose that when the requirements of contact geometry, pressures (stresses), and operating life are considered, optimization of three essential properties of the coatings will provide the ultimate in performance: 1) good coating-substrate adhesion, 2) dense, small grain size (low porosity), and 3) high chemical (phase) purity.

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CONTENTS

FIGURES

TABLES

1. INTRODUCTION

The choice of lubricant for a given application (use) is determined by a number of different factors for the application: the type of contact (rolling or sliding), the contact geometry and pressure, the friction desired, and the lifetime required, to name a few variables. While fluid lubricants remain the ones of choice for many applications, especially those involving high-speed rotation for long periods, there are numerous situations that are best served by the proper choice of solid or dry film lubricant coatings. But, just as the proper base stock, additive package, and formulation must be chosen for conventional fluid lubrication, so should the proper application (deposition) procedure, composition, film structure, and substrate surface be selected for dry film lubrication. It is imperative to understand the relationships between fundamental materials properties and tribological performance in order to select the optimal coating for a specific use. Once these relationships are understood, the details of preparation (deposition conditions) become simply a means of achieving the desired materials properties. A coating could be deposited by sputtering, evaporation, spraying, or any other method if the required properties were obtained.

In the case of MoS_2 coatings, a variety of plasma deposition processes have been used successfully to obtain very low friction coatings with good endurance properties. These processes include dc, rf, rf magnetron sputtering, ion beam assisted deposition (IBAD), post-deposition ion mixing or implantation, and laser ablation or processing. Various parametric studies have been done with each type of deposition, and optimum operating conditions (knob settings) have been identified for lowest friction and lowest wear rates. One of the purposes of this report is to show that such parametric studies resulted in the (perhaps inadvertent) optimization of microstructures, adhesions, and compositions of the coatings for the particular type of test or application performed. Results of materials characterization studies are described and compared to film performance rankings based on specimen tests. Extrapolations to ball bearing applications are made and it is proposed that when the requirements of contact geometry, pressures (stresses), and operating life are considered, the following three essential properties of the coatings will provide the ultimate in performance:

(a) Good coating-substrate adhesion

(b) Dense, small grain size (low porosity)

(c) High chemical (phase) purity.

(In cases where resistance to environmental attack is more important than long operating life, large crystallite and grain size provide better overall performance.)

2. EXPERIMENTAL

MoS_2 films on 440C steel substrates were prepared in three different laboratories by different sputtering techniques (Ref. 1):

(a) rf diode sputtering (rf)

(b) dc triode sputtering (dc)

(c) rf magnetron sputtering (rfm).

The growth conditions for these films have been described in detail elsewhere (Refs. 2, 3, 4). Primary differences involve substrate temperature (rf 70 to 200°C, dc 130 to 175°C, rfm 24 to 70°C) and growth rate (rf 24.5 to 34.5 nm/min, dc 60.0 nm/min, rfm 40 to 60 nm/min). Sputter-etch cleaning of the substrates was done prior to deposition for the dc and rfm films but not for the rf films. For the TEM (transmission electron microscopy) measurements, amorphous carbon substrates were used on standard copper TEM grids, and the films were of the order of 9 to 40 nm thick (Ref. 5). The films for all other testing and analyses were from 500 to 1000 nm thick.

Scanning electron microscopy (SEM) measurements were made by first overcoating the films with a thin gold layer and then using a Cambridge model S 200 microscope (Ref. 6). The gold layer facilitates high resolution microscopy of the samples. A Phillips model EM 420 TEM/STEM with a beam voltage of 120 keV was used for investigation of the film nanostructure, primarily in the TEM mode. Details of the measurements are given in Ref. 5.

Scratch-type stylus measurements and indentations were done with a Sloan Dektak II surface profiling device with careful attention to the load placed on the stylus (Ref. 2). Other indentations were done with a Rockwell "C" diamond brale stylus (Refs. 1, 2).

X-ray photoelectron spectroscopy (XPS) measurements were made with a Surface Science Laboratories "top hat" small spot system equipped with a heatable sample stage (temperature capability up to 1000°C) (Ref. 7). X-ray diffraction (XRD) measurements were made with a Phillips Electronics model APD-3720 vertical diffractometer equipped for normal θ to 2θ scans using Cu Kα X rays (0.154 nm). The orientation was such that only crystallographic planes oriented parallel to the substrate surface produced reflections (Ref. 2).

3. RESULTS AND DISCUSSION

3.1 STRUCTURAL PROPERTIES

The surface topographies of various sputter-deposited MoS_2 films are shown in Fig. 1. These micrographs show that dramatically different appearances can be obtained, depending on conditions during growth. Such variations have been observed by others (Refs. 8, 9) and can be related to the zone model categorization of the films (Ref. 10) as will be shown shortly. The shapes of the particles range from needle-like "worms" to dome-shaped "cauliflower" tops. Also shown in the micrographs are stylus tracks produced with a load of 10 mg on a 12.5 μm diameter tip. The deformations shown in these photos depend on the packing densities or porosities of the respective films, which in turn are related to the bulk film morphologies and nucleation properties. The films deposited at higher temperatures (rf, 200°C, and dc, 130° to 175°C) show the greatest degree of deformation, while rfm films deposited at the lowest temperature (24° to 70°C) (dome-capped topography) show the least deformation. Deposition temperature is perhaps the most obvious variable among these film preparation parameters but, as will be discussed later, it is not the only variable that influences morphology and porosity.

The different surface topographies (morphologies) result from differences in the growth patterns of the films as shown in Fig. 2. The rf and dc films consist of anisotropic platelets that grow in columns perpendicular to the substrate surface plane. According to the structure-zone models for classifying films (Refs. 11, 12), these fall into zone 2, which results when diffusion of the impinging flux along the surface of the substrate dominates growth kinetics. The rfm films initially deposited exhibit a poorly defined fibrous interior which is known as zone 1 and prevails when adatom mobility (surface diffusion) is low, as in the case of low temperature, high growth rate deposition, or in the case of extremely high nucleation site densities with high impurity levels. Rfm films deposited chronologically later (i.e., several months) exhibited a zone 2

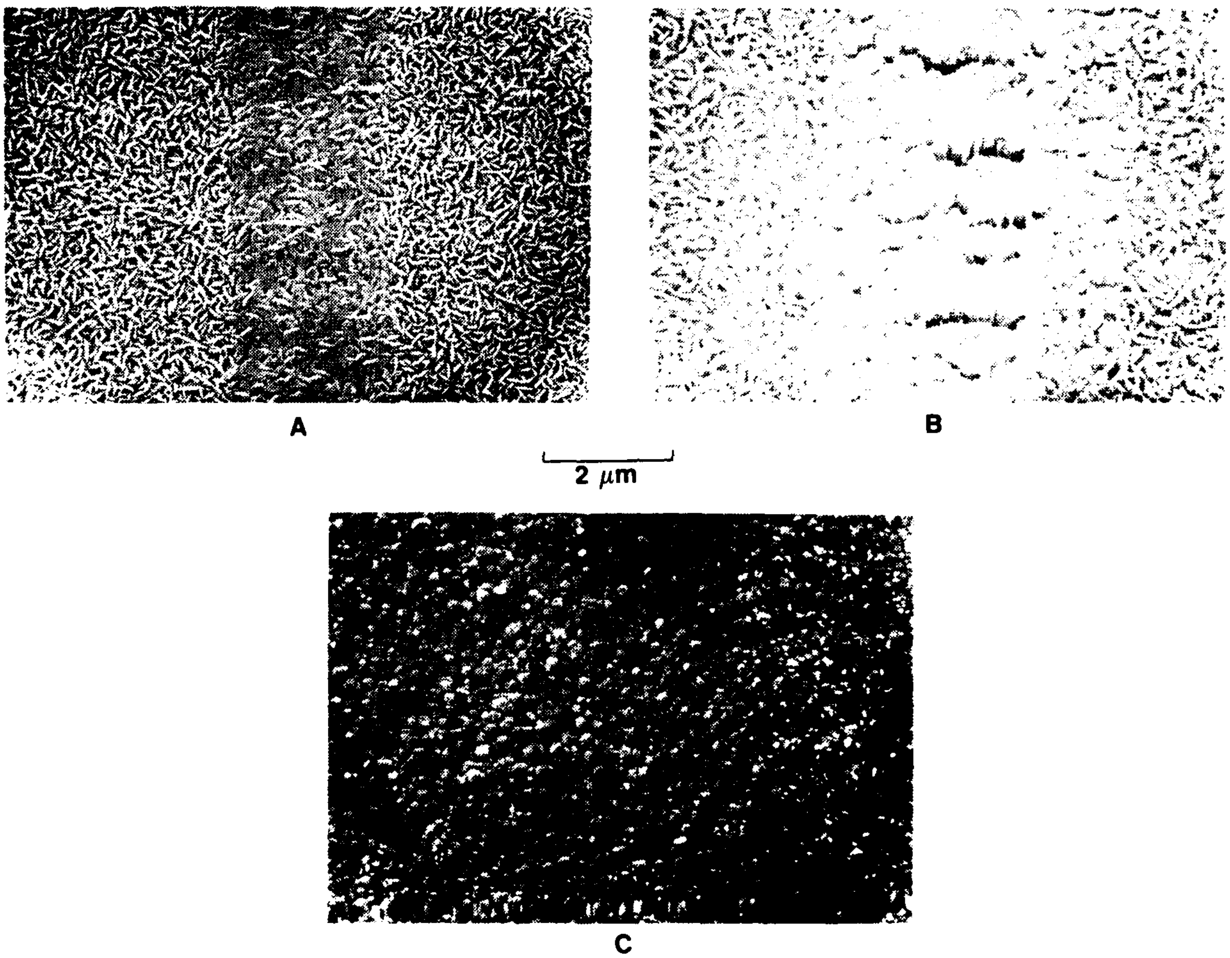

Fig. 1. SEM Micrographs Showing the Extent of Surface Deformation of Various Films After a Stylus Traverse. (A) Rf HT Zone 2, (B) Dc Zone 2, (C) Rfm Zone 1. The dense zone 1 morphology shows the least deformation while the most porous film (dc zone 2) deforms more than the others.

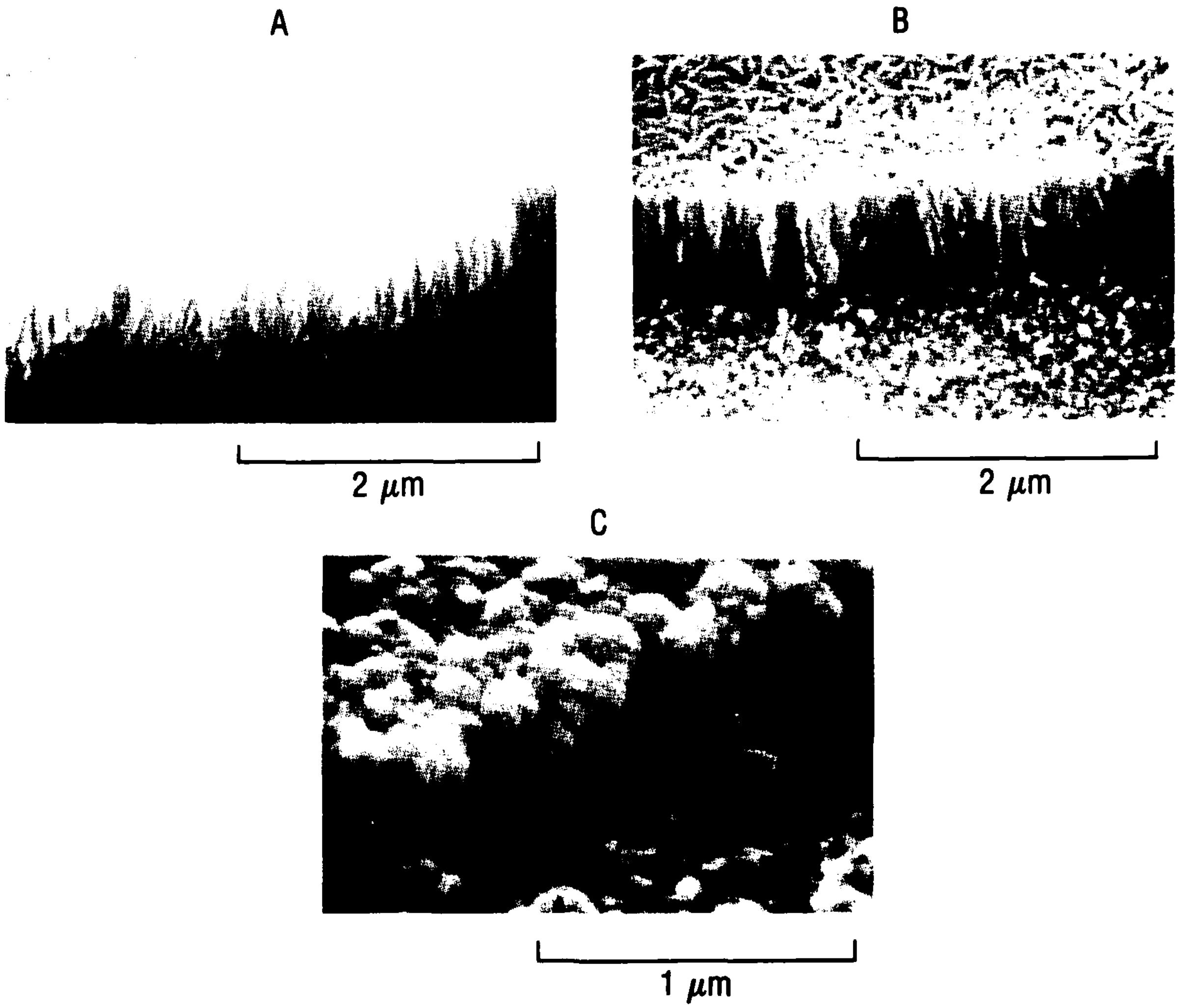

Fig. 2. Cross-Sectional SEM Micrographs Showing Representative Film Morphologies. (A) rf AT Zone 2, (B) dc (Ni) Zone 2, (C) rfm Zone 1.

morphology, similar to the rf and dc films. In addition to the qualitative differences in these film structures, differences in deposition temperature cause variations in particle or platelet size for the zone 2 films. For substrate temperatures of 150° to 200°C, films have relatively large platelets (lengths in the plane of the substrate of approximately 600 to 800 nm), while ambient-temperature (<70°C) films have comparable dimensions of 200 to 400 nm. The dc films also have larger platelets than rf films deposited at approximately the same temperature. Differences in platelet size and spacing between platelets are reflected in different values of measured densities of the films (see Table 1).

Table 1. Density Data

rfm zone 1*	4.07 ± 0.48
rfm zone 2	1.80 ± 0.18
dc zone 2	0.77 ± 0.07
rf zone 2 AT	3.03 ± 0.42
rf zone 2 HT	2.05 ± 0.20
MoS_2 Crystal	4.8
*Of the two groups of rfm zone 1 films, this group had the best properties.	

Surface deformation of film platelets is indicated in Fig. 1 for the sliding contact of a stylus. Films have also been subjected to the sliding contact of a rotating thrust washer in an apparatus described elsewhere (Ref. 2). Cross-sectional views of films in regions of the wear tracks from the washer, showing the type of deformation produced at the outer contact surfaces of the film, are presented in Fig. 3. For zone 2 films (rf and dc) this deformation produces an orientation change in the platelets and crystallites of the films in a thin surface layer as indicated in the micrograph (Fig. 3A) and the accompaning XRD peaks (Fig. 3B). The unworn films have platelets perpendicular to the plane of

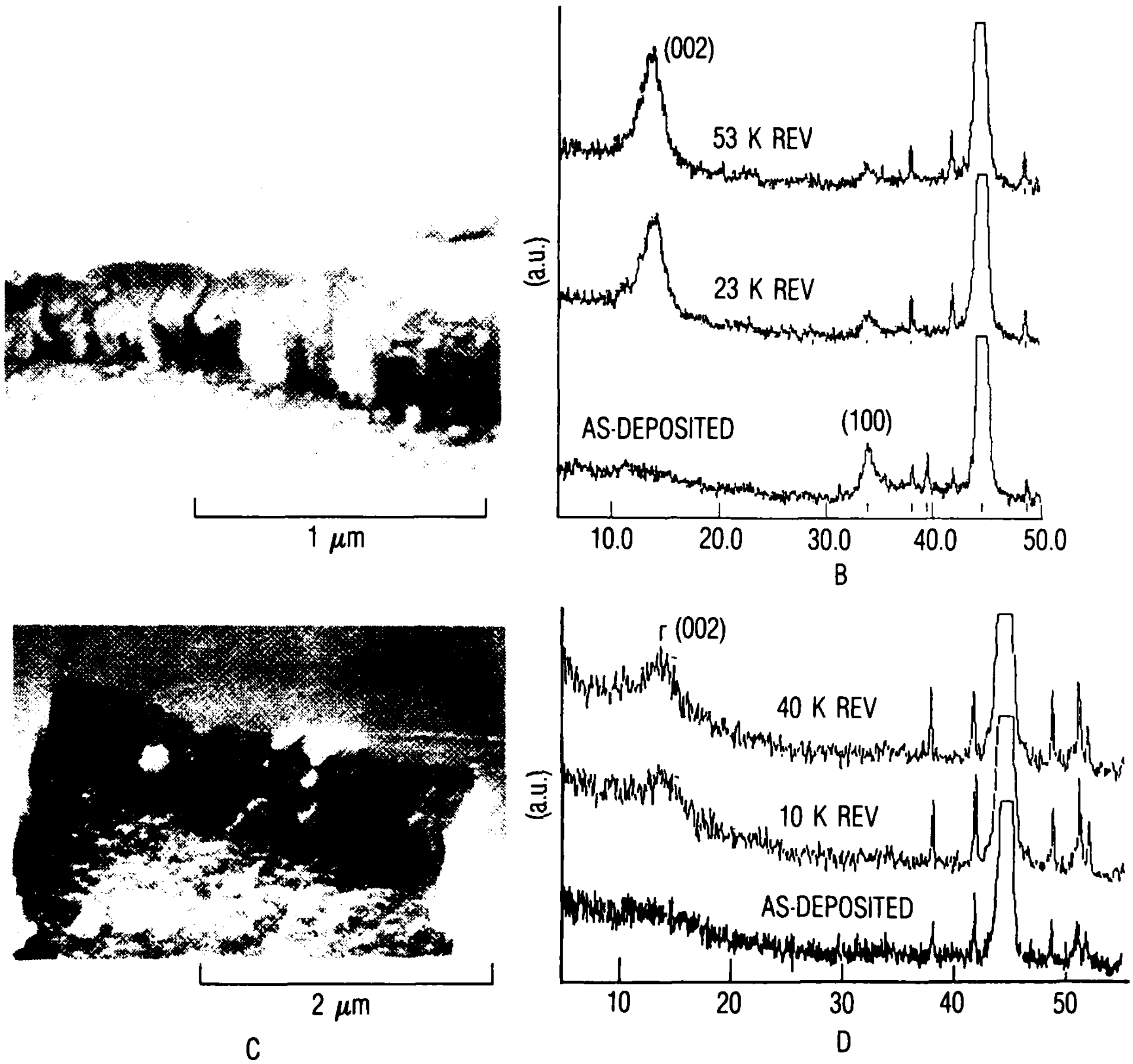

Fig. 3. (A) Cross-Sectional SEM Micrograph Showing That Sliding Wear Deformation Can Be Confined to the Surface of Zone 2 Films. (B) Corresponding X-ray Diffraction Data Which Show That the Deformation Results in a Crystal Reorientation From (100) [and (110), approx. 60°, not shown] to (002) Basal Orientation Parallel to the Surface. (C) Cross-Sectional Micrograph Showing That Sliding Wear Deformation can be Confined to the Surface of Zone 1 Films. (D) Corresponding X-ray Diffraction Data Which Shows That the Deformation Results in a Stress-Induced Crystallization to (002) Basal Orientation Parallel to the Surface.

the substrate surface and XRD peaks for the (100) and (110) edge crystallographic planes with no (002) basal plane peaks. The (002) basal reflection is present for the worn (deformed) film (Fig. 3B), indicating that the orientation of the crystallites in the thin deformed layer has changed from being perpendicular to the surface plane to being parallel (this will be referred to as a change from edge orientation to basal orientation). For the rfm zone 1 films (Figs. 3C,3D), there is no long range crystallinity as shown by the absence of XRD peaks before sliding, but there is induced basal orientation in the deformed layer.

The depth (thickness) of the deformed layer depends on the magnitude of the pressure in the contact region (load) and on properties of the films, such as the platelet size and packing density, the adhesion of the base of the platelets to the substrate, and the coefficient of friction. Films with large platelets and low densities (high degrees of porosity) exhibit deep deformation layers compared to small-platelet, low-porosity films. Consequently, if all other properties are equal (e.g., strengths of adhesion) the wear rates for the former films in applications where the film wear debris is not trapped in the contact area (e.g., a pin-on-disk test) are higher. This was shown in tests of rfm zone 2 films compared to dc zone 2 films (Ref. 13).

3.2 FILM-SUBSTRATE ADHESION

Another very important material property is the strength of adhesion of the film to the substrate. Chemical bonding between the film constituents and the substrate atoms has a strong influence on film growth patterns and adhesion (Ref. 14). If chemical bonding occurs between depositing film atoms and substrate atoms, edge orientation is obtained. The higher the surface density of bonding sites on the substrate surface, the greater will be the number of nucleation sites for film growth and the greater the surface density of platelets within the film. Therefore, the preparation of the substrate surface prior to film deposition will have a major influence on both the adhesion and the porosity of the films. One technique used for preparing substrates is to sputter-etch clean by negatively biasing the substrate and sputtering away surface layers. Typically, a time is chosen for the cleaning process that usually results in superior performance of the film. To our knowledge, it has not been shown before whether the improved performances measured are due to better chemical adhesion or to benefits due to increased surface roughness (Ref. 13).

Figure 4 shows results of brale indentations of rfm films with normal sputter-etch cleaning and with a very limited amount of etching. The degree of cracking and delamination of the film around the edge of the indentation crater can be related to the fracture toughness of the film which depends, in part, on the strength of adhesion (Ref. 1). The results show that the film with minimal pre-etching shows significantly more cracking, which we interpret to mean that its adhesion is poorer than the film with the normal amount of etching. The relative contributions of the surface chemistry and the roughness are not fully understood, and more work is in progress to resolve this issue.

As stated, the orientations of the deposited films and the densities of the nucleation sites are determined, at least partially, by the surface concentrations of chemically active sites. Figure 5 shows why such surface activity has a strong influence on the porosities and, ultimately, on the load carrying capacities of the films. These TEM images of very thin films show that the initial growth stages consist of regions of both edge- and basal-oriented islands. The bright field (BF) image shows the island structure of the film, while the dark field (DF) images created from portions of diffracted electron beams for different crystallographic planes show the regions of either edge (Fig. 5B) or basal (Figs. 5C, 5D) islands (Ref. 5). (The orientation of the electron diffraction is opposite to that of X-ray diffraction so that when an image is observed it means that the diffracting plane is perpendicular to the view plane, which in this case is the substrate plane.) For this film there are large regions of basal-oriented material between the edge-oriented islands, the latter being the more strongly bound. As more material would impinge on this structure during the growth of a thicker film, the edge islands would grow out from

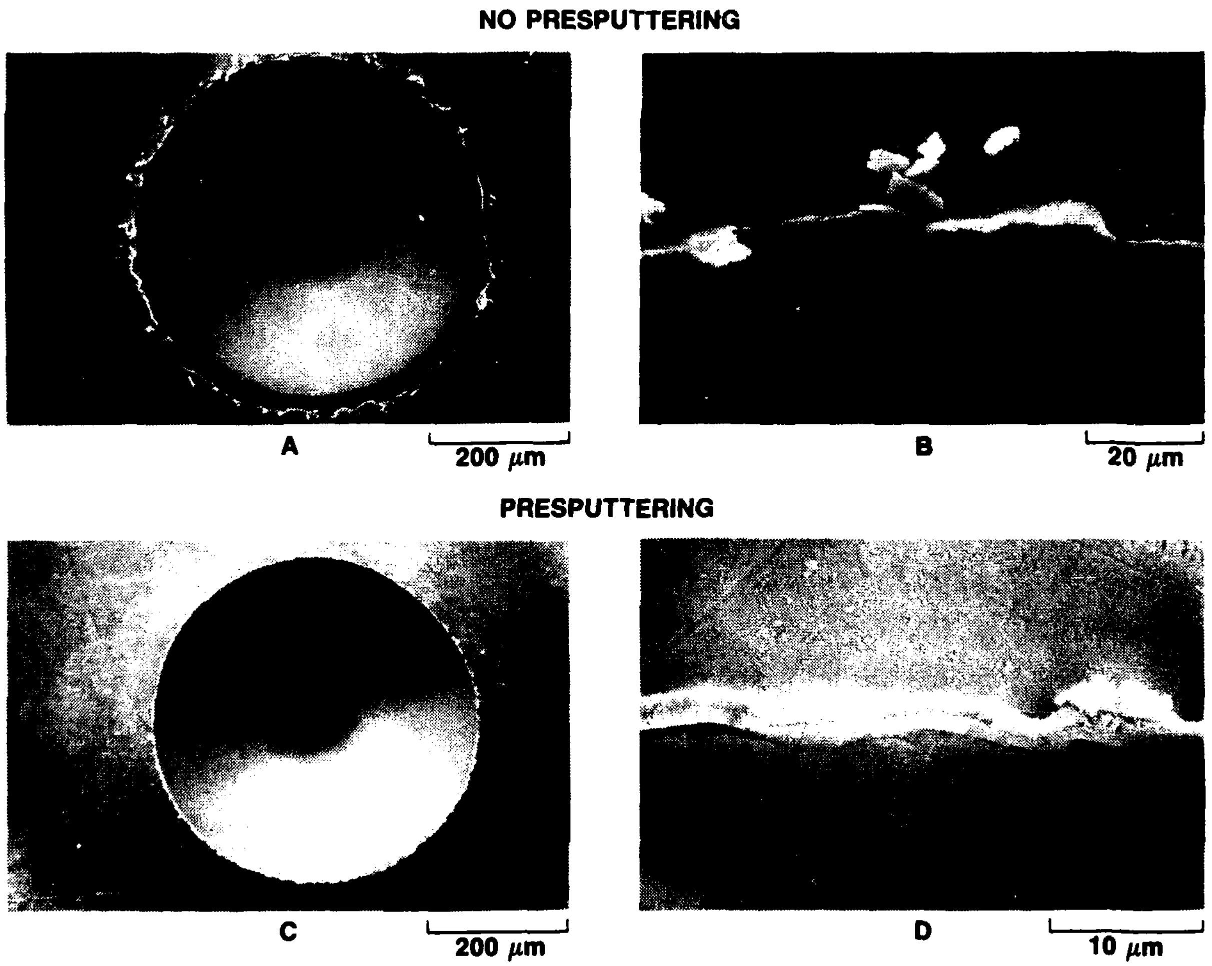

Fig. 4. SEM Micrographs of Rfm Zone 2 Films That are One Micron Thick as a Function of Presputter Time. (A, B) Minimum Sputter Pretreatment Where Significant Delamination Occurs. (C, D) Normal Pretreatment in Which Delamination is Inhibited.

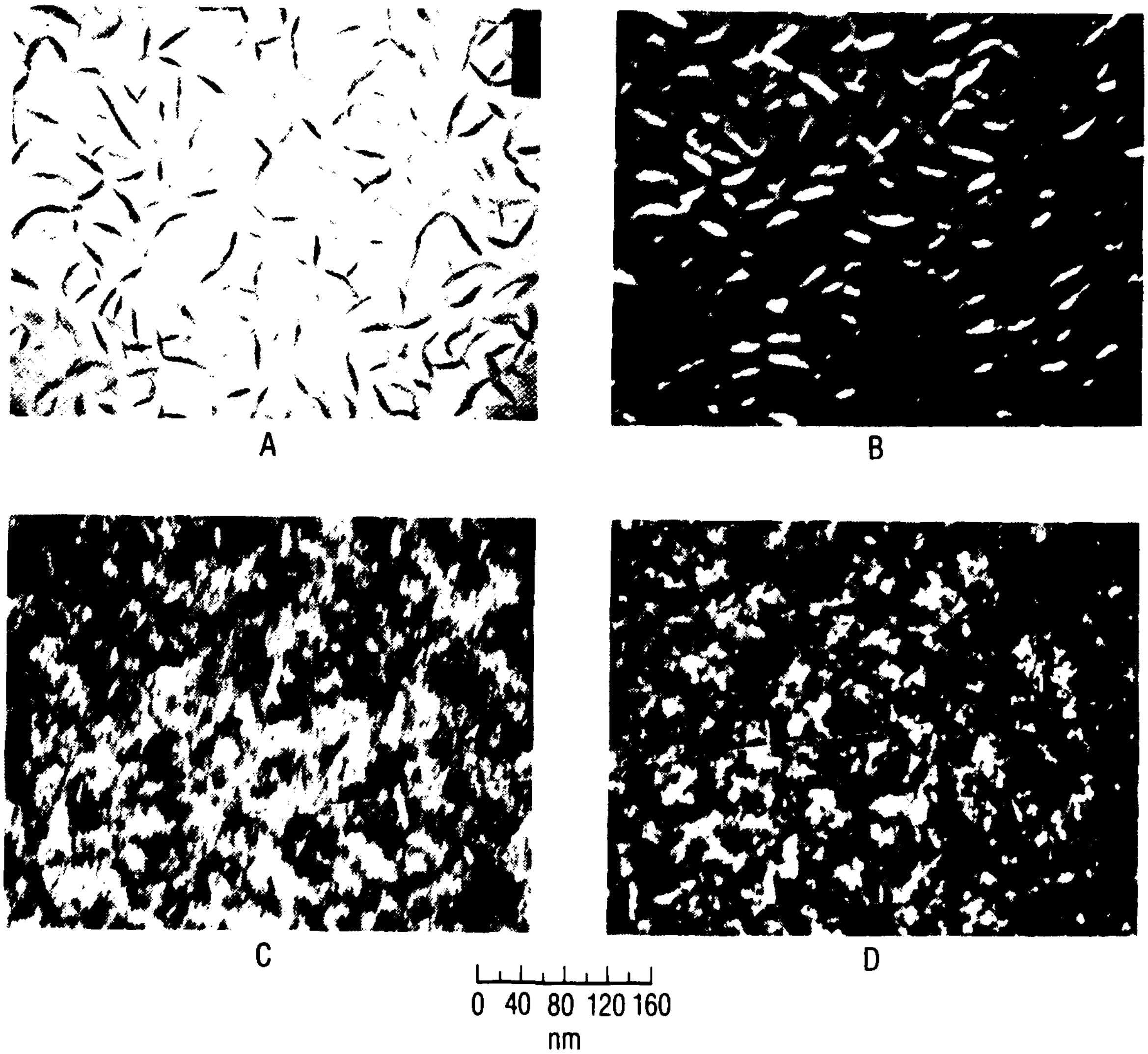

Fig. 5. TEM Micrographs of an Rf HT Zone 2 MoS_2 Film on Amorphous Carbon (Film Thickness Approx. 40 nm). (A) Bright-Field (BF) image, (B) A Corresponding (002) Dark-Field (DF) Image, (C) A (100) Dark-Field Image, and (D) A (110) Dark-Field Image. The images show that basal orientation perpendicular to the substrate is confined to the acicular "edge" islands and that edge-plane orientation perpendicular to the substrate is more widespread, indicating the existence of "basal" islands in which the (001) basal orientation is parallel to the substrate between the edge islands.

the surface more rapidly because of the higher reactivity of the edge plane surfaces. However, these growing platelets would be widely separated at their points of connection to the substrate and would be expected, therefore, to have relatively open (porous) structures and low fracture toughnesses. Consequently, surface pretreatments that increase the density and chemical reactivity of nucleation sites on the substrate surface serve two purposes:

(a) To increase the film to substrate adhesion

(b) To increase the packing density (decrease porosity) of the columns comprising the films.

3.3 CHEMICAL COMPOSITIONS

The third important materials parameter, besides structure and adhesion, is the chemical composition of the deposited film. The purities of sputter-deposited films vary widely from being sulfur-deficient to the opposite, sulfur enrichment, and measurements have shown that the basic molybdenite structure can be obtained even for films with S to Mo ratios as low as 1.1 (Ref. 15). Oxygen is often a major contaminant of films and can be present in a variety of chemical forms. A significant recent finding is that in addition to the often seen MoO_3 contaminant in sputter-deposited films, oxygen can substitute for sulfur in the MoS_2 lattice (Ref. 7). Films were annealed at different temperatures, up to 700°C, and the atomic ratio of (S + O) to Mo was found to remain at 2 after the initial adsorbed oxygen was desorbed. The conclusion of this work was that a phase, $MoS_{2-x}O_x$, was present and that it is this phase that is probably the important constituent for good lubrication. Further, the presence of oxygen in the form of MoO_3 is uniformly detrimental to film performance, in terms of both friction and endurance. Some oxygen content in the $MoS_{2-x}O_x$ phase is believed to be good for producing lower friction and longer endurance (Ref. 16) films.

3.4 WEAR TESTING

Specimen wear tests involving moderate contact stress (700 MPa, pin-on-disk) with expulsion of wear debris from the contact area show that film-substrate adhesion is the most critical parameter in determining endurance life (Table 2), comparing the results for rf films with no sputter-etch cleaning to those for rfm and dc. For films with comparable adhesion and similar morphologies (dc and rfm zone 2), those with smaller grain size have greater endurance in pin-on-disk tests. Changing the contact geometry to the line contact of the rub-shoe tester results in some degree of debris retention such that larger grain size is not detrimental, but better adhesion still gives better endurance. Finally, going to the thrust washer with low contact stress results in trapping practically all of the film debris and makes adhesion practically unimportant as long as the films do not delaminate. For this latter case, compact packing (low porosity) appears to be more important than adhesion.

Table 2. Wear Test Results

Zone	Pin-On-Disk	Dual-Rub-Shoe	Thrust-Washer
rfm zone 1	201 K rev	19 K rev[a]	600–700 K rev
rfm zone 2	—	55 K rev[c]	800 K rev
dc zone 2	156 K rev	60 K rev[b]	300 K rev
rf zone 2 AT	45 K rev	35 K rev[a]	900 K rev

[a]Tested in air; [b]Air or vacuum; [c]Vacuum.

4. CONCLUDING REMARKS

A summary of the conclusions of this work is provided in Table 3. Three primary materials properties: composition or phase purity, adhesion (fracture toughness), and morphology and porosity are believed to be the primary drivers of the three important performance parameters of friction, endurance, and debris generation, respectively. Crystallinity and orientation are related to morphology, and substrate - counterface compositions are involved in adhesion, but also can have an independent role in determining friction.

Table 3. Performance Parameters and Materials Properties of MoS_2 Lubricating Films

	Friction	Endurance	Debris
Composition Phase Purity	1	2	
Adhesion Fracture Tough		1	2
Morph./Porosity "Softness"		2	1
Crystallinity Orientation	2		
Substrate-Counterface Composition	2	2	

1 = Primary Determinant, 2 = Secondary Determinant of Performance.

The choice of the proper MoS_2 film for a specific use depends on the contact geometry, stress levels, and a trade between operational life and environmental factors, both in storage and during operation. The ideal combination of good adhesion, chemical purity, and dense, small-grain size in MoS_2 films provides for the best lubrication and endurance in most relatively high-contact stress conditions with moderate operating lifetimes (10^8 to 10^{10} revolutions), such as for torque sensitive ball bearings. This combination of optimal film properties can be obtained by high-rate, ambient temperature (<70°C) sputtering with substrate surface etching prior to sputtering. Optimal properties can also be obtained by post-deposition ion processing with high energy ions (≥ 100 keV) or by low energy (1 to 2 keV) ion processing during deposition. Other applications such as telescoping joints, release mechanisms, actuators, and solar array drive mechanisms may not require the optimal endurance but may need protection from environmental factors. Ultra-low friction can be obtained with resistance toward oxidation (to form MoO_3) by using growth conditions (usually higher temperatures) that induce larger crystallite and grain formation. Such films generate relatively large wear particles during operation that can cause noise in torque sensitive applications and thus are recommended only if torque stability is not critical.

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